



## PARTIAL ENGLISH TRANSLATION OF JP-A-61-171737

· page 2, upper, left col. line 9 ~ page 2, upper, right col. line 13

[Means for solving the Problem] and [Function]

The present invention relates to a process for crosslinking an elastomer, where the elastomer is crosslinked by subjecting rubber to primary crosslinking by sulfur or a sulfur-based compound, and then to secondary crosslinking by radio active rays.

Crosslinkable elastomer includes natural rubber or synthetic rubber, for example, styrene-butadiene rubber, acrylonitrile-butadiene rubber, hydrogenated acrylonitrile-butadiene rubber, chloroprene rubber, epichlorohydrin rubber, ethylene-propylene rubber, active chlorine-containing acrylic rubber, chlorosulfonated polyethylene, chlorinated polyethylene, butyl rubber, silicone rubber, active halogen-containing fluororubber, etc., as crosslinked by sulfur or a sulfur-based compound.

The sulfur-based compound for use in primary crosslinking like sulfur includes imidazoles typically, e.g. 2-mercaptoimidazole, thioureas typically, e.g. N,N' -diethylthiourea, thiazoles typically, e.g. mercaptobenzothiazole, and dibenzylthiazyl disulfide, dicarbamic acid salts, thiurams, and their mixtures. Primary crosslinking can be carried out by the sulfur-based crosslinking agent under the ordinary crosslinking conditions.

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[EXAMPLES]

The present invention will be described below, referring to Examples.

## COMPARATIVE EXAMPLES 1 AND 2

<u>Compounding component</u>	Comp.	Comp.
	<u>Ex. 1</u>	<u>Ex. 2</u>
Hydrogenated acrylonitrile-butadiene rubber (Zetpol 2020, a product of Japan Zeon Co.)	100	100
Zinc oxide	3	3
Magnesium oxide	7	7
Stearic acid	2	2
FEF carbon black	50	50
2-mercaptobenzoimidazole	2	2
Di-t-butylperoxydiisopropylbenzene (concentration : 40%)	—	6
Triallyl isocyanurate (concentration : 70%)	—	2
Tetramethylthiuram disulfide	1.5	—
Tetraethylthiuram disulfide	1.0	—
Mercaptobenzothiazole	0.5	—
Sulfur	0.5	—

Compositions in the foregoing compounding proportions (parts by weight) was subjected to primary crosslinking at 180°C for 5 minutes, and then to secondary crosslinking at 150° for 3 hours. Primary crosslinking products and secondary crosslinking products of Comparative Example 1 (sulfur-based crosslinking) and Comparative Example 2 (peroxide-based crosslinking) were each subjected to determination of physical properties according to JIS K-8301.

## EXAMPLE 1

In Comparative Example 1, the secondary crosslinking was carried out by irradiation of 20-megarad  $\gamma$ -rays of Co 60.

Results of determination obtained in the foregoing Comparative

Examples and Example are shown in the following Table 1.

Table 1

<u>Item of determination</u>	<u>Comp. Ex.1</u>	<u>Comp. Ex.2</u>	<u>Ex.1</u>
[Primary crosslinking products]			
Hardness (degree)	75	77	78
Tensile strength (MPa)	26.2	26.8	26.2
Elongation (%)	461	283	461
[Secondary crosslinking products]			
Hardness (degree)	81	80	82
Tensile strength (MPa)	24.6	27.2	27.0
Elongation (%)	269	246	185
Compression set (150°C for 70 hrs) (%)	83	28	32

COMPARATIVE EXAMPLES 3 AND 4

<u>Compounding components</u>	<u>Comp. Ex.3</u>	<u>Comp. Ex.4</u>
Ethylene-propylene-diene terpolymer rubber (EPT 1070, a product of Mitsui Petrochemical Co.)	100	100
FEF carbon black	80	80
Wax	2	2
Synthetic polyterpene resin	6	6
Zinc oxide	5	—
Substituted diphenyl amine	—	2
2,2,4-trimethyl-1,2-dihydroquinoline polymer	2	—
Dicumyl peroxide	—	5
Triallyl isocyanurate (concentration : 70%)	—	3
Sulfur	1.5	—
Tetramethylthiuram disulfide	1.5	—
2-mercaptobenzothiazole	0.3	—

Compositions in the foregoing compounding proportions (parts by weight) were subjected to primary crosslinking at 180°C for 5 minutes, and then to secondary crosslinking at 150°C for 5 hours. Primary crosslinking products and secondary crosslinking products of Comparative Example 3 (sulfur-based crosslinking) and Comparative Example 4 (peroxide-based crosslinking) were each subjected to determination of physical properties according to JIS K-8301 and also to determination of percent changes in physical properties, when heated at 120°C or 150°C for 280 hours as a measure of heat resistance of the secondary crosslinking products.

#### EXAMPLE 2

In Comparative Example 3, the secondary crosslinking was carried out by 15-megarad  $\gamma$ -rays of Co 60.

Results of determination obtained in Comparative Examples 3 and 4, and Example 2 are shown in the following Table 2.

Table 2

<u>Item of determination</u>	<u>Comp. Ex.3</u>	<u>Comp. Ex.4</u>	<u>Ex. 2</u>
[Primary crosslinking products]			
Hardness (degree)	75	76	75
Tensile strength (MPa)	9.5	11.4	9.6
Elongation (%)	350	240	350
[Secondary crosslinking products]			
Hardness (degree)	81	78	79
Tensile strength (MPa)	13.2	13.5	12.5
Elongation (%)	220	210	180
Compression set (150°C for 70 hrs) (%)	42	15	18
[Heat resistance : 120°C for 280 hrs]			
Hardness change (degree)	+5	+3	+3

Tensile strength change	(%)	+2	+3	+5
Elongation change	(%)	-20	-15	-15
[Heat resistance : 150°C for 280 hrs]				
Hardness change	(degree)	+8	+5	+5
Tensile strength change	(%)	+10	+7	+6
Elongation change	(%)	-35	-25	-30

## COMPARATIVE EXAMPLE 5

Parts by weight

Active chlorine-containing acrylic rubber	100
(Noxtime PA-402, a product of Nippon Mectron Co.)	
FEF carbon black	60
Stearic acid	1
Substituted diphenyl amine	2
Sodium stearate	4
Sulfur	0.3
Trimethylolpropane trimethacrylate	2

Composition in the foregoing compounding proportions was subjected to primary crosslinking at 180°C for 5 minutes, and then to secondary crosslinking at 150°C for 5 hours. The resulting secondary crosslinking product was subjected to determination of physical properties according to JIS K-8301.

## EXAMPLE 3

In Comparative Example, the secondary crosslinking was carried out by irradiation of 25-megarad  $\gamma$  rays of Co 60.

Results of determination obtained in Comparative Example 5 and Example 3 are shown in the following Table 3.

Table 3

<u>Item of determination</u>		<u>Comp. Ex. 5</u>	<u>Ex. 3</u>
Hardness	(degree)	70	72
Tensile strength	(MPa)	13.2	14.3
Elongation	(%)	180	150
Compression set (150°C for 70 hrs)	(%)	35	28